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(54) PAPIER A CAPACITE DE TRANSFORMATION ACCRUE FAIT AVEC DES AGENTS DE COLLAGE DE TYPE 2-OXETANONE

(54) SYNTHESIS OF ALKYL KETENE MULTIMERS (AKM) AND APPLICATION FOR PRECISION CONVERTING GRADES OF FINE PAPER

(57) Disclosed is a process for making fine paper that is sized under alkaline conditions with a 2-oxetanone sizing agent. The sizing agent is a mixture of alkyl ketene dimer and 2-oxetanone multimers of various molecular weights, at least 50% by weight of the 2-oxetanone compounds having at least two 2-oxetanone rings. The paper can be in the form of continuous forms bond paper, adding machine paper, and reprographic paper and can also be converted into envelopes. The paper exhibits levels of sizing comparable to those obtained with current alkyl ketene dimer and alkenyl succinic anhydride sizes, and gives improved performance in high speed converting and reprographic machines.

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PATENT

Bottorff & Zhang Case 1

CA2117318

PAPER WITH IMPROVED CONVERSION CAPABILITY MADE WITH 2-OXETANONE SIZING AGENTS

Abstract of the Disclosure

Disclosed is a process for making fine paper that is sized under alkaline conditions with a 2-oxetanone sizing agent. The sizing agent is a mixture of alkyl ketene dimer and 2-oxetanone multimers of various molecular weights, at least 50% by weight of the 2-oxetanone compounds having at least two 2-oxetanone rings. The paper can be in the form of continuous forms bond paper, adding machine paper, and reprographic paper and can also be converted into envelopes. The paper exhibits levels of sizing comparable to those obtained with current alkyl ketene dimer and alkenyl succinic anhydride sizes, and gives improved performance in high speed converting and reprographic machines.

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This invention relates to paper containing alkaline sizing agents, and processes for using the paper.

The amount of fine paper produced under alkaline conditions has been increasing rapidly, encouraged by cost savings, the ability to use precipitated calcium carbonate, an increased demand for improved paper permanence and brightness, and an increased tendency to close the wet end of the paper machine.

Current applications for fine paper require particular attention to sizing before conversion or end-use, such as high-speed photocopies, envelopes, forms bond including computer printer paper, and adding machine paper. The most common sizing agents for fine paper made under alkaline conditions are alkenyl succinic anhydride (ASA) and alkyl ketene dimer (AKD). Both types of sizing agents have a reactive functional group that covalently bonds to cellulose fiber and hydrophobic tails that are oriented away from the

fiber. The nature and orientation of these hydrophobic tails cause the fiber to repel water.

Commercial AKD's, containing one ß-lactone ring, are prepared by the dimerization of the alkyl ketenes made from two saturated, straight-chain fatty acid chlorides; the most widely used being prepared from palmitic and/or stearic acid. Other ketene dimers, such as the alkenyl-based ketene dimer (Aquapel® 421, available from Hercules Incorporated, Wilmington, DE, U.S.A.), have also been used commercially. ASA-based sizing agents can be prepared by the reaction of maleic anhydride with a 14 to 18 carbon olefin.

Although ASA and AKD sizing agents are commercially successful, they have disadvantages. Both types of sizing agents, particularly the AKD type, have been associated with handling problems in the typical high-speed conversion operations required for the current uses of fine paper made under alkaline conditions (referred to as alkaline fine paper). The problems include reduced operating speed in forms presses and other converting machines, double feeds or jams in high-speed copiers, and registration errors on printing and envelope-folding equipment that operate at high speeds.

These problems are not normally associated with fine paper produced under acid conditions (acid fine paper). The types of filler and filler addition levels used to make alkaline fine paper differ significantly from those used to make acid fine paper, and can cause differences in paper properties such as stiffness and coefficient of friction, which affect paper handling. Alum addition levels in alkaline fine paper, which contribute to sheet conductivity and dissipation of static charge, also differ significantly from those used in acid fine paper. This is important because the electrical properties of paper affect its handling performance. Sodium chloride is often added to the surface of alkaline fine paper to improve its end-use performance.

The typical problems encountered with the conversion and end-use handling of alkaline fine paper involve:

1. Paper properties related to the composition of the furnish;

2. Paper properties developed during paper formation; and

3. Problems related to sizing.

The paper properties affected by papermaking under alkaline conditions that can affect converting and end-use performance include:

- Curl
- Variation in coefficient of friction
- Moisture content
- Moisture profile
- Stiffness
- Dimensional stability
- MD/CD strength ratios

One such problem has been identified and measured as described in "Improving the Performance of Alkaline Fine Paper on the IBM 3800 Laser Printer," TAPPI Paper Makers Conference Proceedings (1991). The problem occurs when using an IBM 3800 high speed continuous forms laser printer that does not have special modifications intended to facilitate handling of alkaline fine paper. This commercially-significant laser printer therefore can serve as an effective testing device for defining the convertibility of various types of sized paper on state-of-the-art converting equipment and its subsequent end-use performance. In particular, the phenomenon of "billowing" gives a measurable indication of

the extent of slippage on the IBM 3800 printer between the undriven roll beyond the fuser and the driven roll above the stacker.

Such billowing involves a divergence of the paper path from the straight line between the rolls, which is two inches (5 cm) above the base plate, causing registration errors and dropped folds in the stacker. The rate of billowing during steady-state running time is measured as the billowing height in inches above the straight paper path after 600 seconds of running time and multiplied by 10,000.

Typical alkaline AKD sized fine paper using a size furnish of 2.2 lbs. per ton (1 kg per 0.9 metric ton) of paper shows an unacceptable rate-of-billowing, typically of the order of 20 to 80. Paper handling rates on other high-speed converting machinery, such as the Hamilton-Stevens continuous forms press, or the Winkler & Dunnebier CH envelope folder also provide numerical measures of convertibility.

Ketene multimers containing more than one beta-lactone ring have also been disclosed as sizing agents for paper in Japanese Kokai 168991/89 and 168992/89. The ketene multimers are said to show improved sizing compared to the ketene

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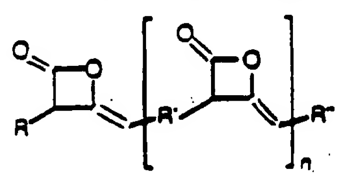
dimers previously used. The multimers are prepared from a mixture of mono- and dicarboxylic acids. The advantage of a combination of good sizing and good performance on high speed converting or reprographic equipment is not disclosed. These references also do not disclose the specific multimers claimed in the present invention.

There is a need for alkaline fine paper that provides improved handling performance in typical converting and reprographic operations. At the same time, the levels of sizing development must be comparable to that obtained with the current furnish levels of AKD or ASA for alkaline fine paper.

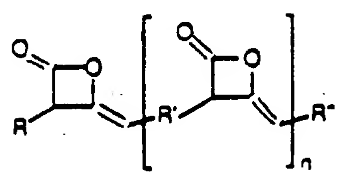
The paper of this invention sized with a sizing agent comprising a mixture of 2-oxetanone compounds is characterized in that the paper is made under alkaline conditions and the 2-oxetanone compounds have the formula



in which n is 0 or an integer; R and R", which can be the same or different, are saturated, linear straight chain alkyl groups having 8-24 carbon atoms, preferably 14 or 16 carbon

atoms; R′ is a saturated, linear alkyl group having 4-40 carbon atoms, preferably 5 or 6 carbon atoms, and in at least 50% by weight of the 2-oxetanone compounds, preferably 60%, n = at least 1. This paper is capable of performing without encountering significant machine-feed problems on high speed converting machines or in reprographic operations.

In a broad aspect, then, the present invention relates to paper sized with a sizing agent comprising a mixture of 2-oxetanone compounds, characterized in that the paper is made under alkaline conditions and the 2-oxetanone compounds have the formula:



in which n is 0 or an integer; R and R" can be the same or different and are saturated, linear alkyl groups having 8-24 carbon atoms; R′ is a saturated, linear alkyl group having 4-40 carbon atoms, and n = at least one in at least 50% by weight of the compounds in the mixture, and the paper does not encounter significant machine-feed problems on high speed converting machines or in reprographic operations.

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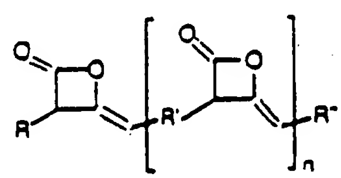
Also according to the invention, the mixture of 2-oxetanone compounds is the reaction product of a reaction mixture comprising at least one 10-26 carbon linear, saturated alkyl monocarboxylic acid and at least one 8-44 carbon linear, saturated alkyl dicarboxylic acid, wherein the mole ratio of mono- to dicarboxylic acids is 1.0 to 3.5, at least 50% by weight of the 2-oxetanone compounds in the mixture having at least two 2-oxetanone rings.

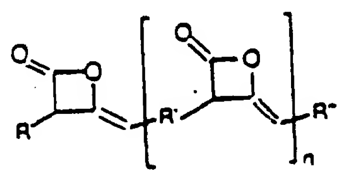
In another broad aspect, then, the present invention relates to a mixture of 2-oxetanone compounds characterized in that the 2-oxetanone compounds have the formula:



in which n is 0 or an integer; R and R" can be the same or different and are saturated, linear alkyl groups having 8-24 carbon atoms, and R′ is a saturated, linear alkyl group having 4-40 carbon atoms, said mixture being the reaction product of a reaction mixture comprising at least one 10-26 carbon linear, saturated alkyl monocarboxylic acid and at least one 8-44 carbon linear, saturated alkyl dicarboxylic acid, wherein the ratio of mono- to dicarboxylic acid is 1.0 to 3.5, and at least 50% by

weight of the compounds in the mixture have at least two 2-oxetanone rings.

In yet another broad aspect, the present invention relates to a process for making paper for performance in precision converting or reprographic machinery, characterized by the step of internally sizing the paper under alkaline conditions with a 2-oxetanone sizing agent comprising a mixture of 2-oxetanone compounds having the formula:



in which n is 0 or an integer, R and R" can be the same or different and are saturated, linear alkyl groups having 8 - 24 carbon atoms; R' is a saturated, linear alkyl group having 4 - 40 carbon atoms, and n = at least one in at least 50% by weight of the compounds in the mixture.

In another broad aspect, the present invention relates to the use of paper sized with a sizing agent comprising a mixture of 2-oxetanone compounds, characterized in that the paper is sized under alkaline conditions; the 2-oxetanone compounds having the formula

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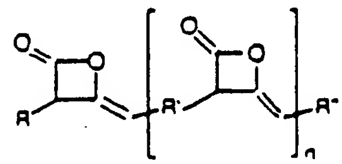

in which n is O or an integer, R and R" can be the same or different and are saturated, linear alkyl groups having 8 - 24 carbon atoms; R' is a saturated, linear alkyl group having 4 - 40 carbon atoms, and n = at least one in at least 50% by weight of the compounds in the mixture, and the paper is used in high speed converting or reprographic operations.

Preferably, the paper according to the invention is capable of being formed into a roll of continuous forms bond paper having a basis weight of 30 to 60 lb/3000 ft² (13.6 to 27.2 kg per 279 m²) and is capable of running on the IBM Model 3800 high speed, continuous-forms laser printer without causing a rate of billowing in inches of increase per second x 10,000 greater than 5.

The paper of this invention can be made into sheets of 8 1/2 x 11 inch (21.6 x 28 cm) reprographic cut paper having a basis weight of 15-24 lb/1300 ft^2 (6.8 to 10.9 kg/121 m^2) and is capable of running on a high speed laser printer or copier without causing misfeeds or jams at a rate of 5 or less in 10,000.

The paper of this invention having a basis weight of 20-24 lb/3000 ft^2 (9.1 to 10.9 kg/279 m^2) can be converted to a standard perforated continuous form on a continuous forms press at a press speed of 1300 to 2000 feet (390 m to 600 m) per minute. The preferred paper according to the invention having a basis weight of 20-24 lb/3000 ft^2 (9.1 to 10.9 kg/279 m^2), and that is sized at an addition rate of at least 2.2 lb/ton (1 kg/0.9 metric ton) can be converted to a standard perforated continuous form on the Hamilton-Stevens continuous forms press at a press speed of at least 1775 feet (541 m) per minute, preferably 1900 feet (579 m) per minute.

Also according to the invention, paper having a basis weight of 20-24 lb/1300 ft^2 (9.1 to 10.9 kg/121 m^2) can be converted into at least 900 envelopes per minute, preferably at least 1000 per minute, on a Winkler & Dunnebier CH envelope folder.

Machine-feed problems on high speed converting machines or during reprographic operations are defined as significant in any specific conversion or reprographic application if they cause misfeeds, poor registration, or jams to a commercially unacceptable degree as will be discussed below, or cause machine speed to be reduced.

The process of this invention for making paper under alkaline conditions exhibits levels of sizing comparable to those obtained with current AKD and ASA sizing technology, and gives improved handling performance in typical end-use and converting operations.

The 2-oxetanone sizing agents of this invention are a mixture of saturated, linear alkyl ketene dimers and 2-oxetanone multimers of varying molecular weights, at least 50 mole %, preferably 60 mole %, of the compounds in the mixture having at least two 2-oxetanone rings. The sizing agent therefore has more reactive sites for covalently bonding with cellulose fibers than conventional AKD and ASA alkaline sizes. The mole % of compounds having two or more 2-oxetanone rings increases as the mole ratio of mono- to dicarboxylic acids decreases.

These reactive sizing agents can be prepared using methods disclosed in Japanese Kokai 168992/89 and U.S.P. 4,317,756. In the first step, acid chlorides are formed from a mixture of at least one linear, saturated alkyl monocarboxylic acid and at least one linear, saturated alkyl dicarboxylic acid, using phosphorous trichloride or another conventional chlorinating agent. The mole ratio of mono- to dicarboxylic acids is 1.0 to 3.5, preferably 2.5, and more preferably 2.0. The acid chlorides are then dehydrochlorinated in the presence of triethylamine or another suitable base, in propylene dichloride or another anhydrous, aprotic solvent, to form the mixture of 2-oxetanone compounds. The monocarboxylic acid can be a mixture of C_{16} and C_{18} linear, saturated alkyl monocarboxylic acids, for example, Emery 135 fatty acids, available from Henkel-Emery, Cincinnati, Ohio, U.S.A. Stable emulsions of these sizing agents can be prepared in the same way as standard AKD emulsions.

The linear, saturated alkyl monocarboxylic acids used to prepare the 2-oxetanone compounds of this invention have 10-26 carbon atoms, preferably 14-22 carbon atoms, and most preferably 16-18 carbon atoms. These acids include, for

example, stearic, myristic, palmitic, margaric, pentadecanoic, decanoic (capric), undecanoic, dodecanoic (lauric), tridecanoic, nonadecanoic, arachidic, and behenic acids. One or more of these monocarboxylic acids can be used.

The linear, saturated alkyl dicarboxylic acids used to prepare the 2-oxetanone multimers of this invention have 8-44 carbon atoms, preferably 9-10, 22 or 36 carbon atoms. Dicarboxylic acids with 9-10 carbon atoms are most preferred. Such dicarboxylic acids include, for example, sebacic, azelaic, dodecanedioic, suberic, brazylic, and docosanedioic acids, and EMPOL 1008 dimer acids (C_{36}), available from Henkel-Emery, Cincinnati, Ohio, U.S.A. One or more of these dicarboxylic acids can be used.

Preferably the alkaline paper made according to the process of this invention contains a water soluble inorganic salt of an alkali metal, preferably sodium chloride (NaCl), as well as alum and precipitated calcium carbonate. However, the paper of this invention will often be made without NaCl.

The sizing agents of this invention can be applied as internal sizing agents or surface sizing agents. Internal sizing involves adding the size to the paper pulp slurry

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before sheet formation, while surface sizing involves immersion of the paper in the sizing agent or spraying the sizing agent on the paper, followed by drying at elevated temperatures using known drying techniques.

The paper of this invention is generally sized at a size addition rate of at least 0.5 lb (0.23kg), preferably at least 1.5 lb (0.68kg), and more preferably at least 2.2 lb/ton (1 kg/0.9 metric tons) or higher. It may be, for example, in the form of continuous forms bond paper, perforated continuous forms paper, adding machine paper, or envelope-making paper, as well as converted products, such as copy paper and envelopes.

The paper of this invention is capable of performing effectively in tests that measure its convertibility on state-of-the-art converting equipment and its performance on high speed end-use machinery. In particular, the paper according to the invention that can be made into a roll of continuous forms bond paper having a basis weight of 30 to 60 lb/3000 ft^2 (13.6 to 27.2 kg/279 m^2), preferably 40 to 50 lb/3000 ft^2 (18 to 22.6 kg/279 m^2), is capable of running on a high speed, continuous forms laser printer. When this paper is sized at an addition rate of at least 2.2 lb/ton (1 kg/0.9

metric ton), it is capable of running on the IBM Model 3800 high speed, continuous-forms laser printer without causing a rate of billowing in inches of increase per second x 10,000 greater than 5 after ten minutes running time, preferably 3 or less.

Further, the preferred paper according to the invention, that can be made into sheets of 8 1/2 x 11 inch (21.6 cm x 28 cm) reprographic cut paper having a basis weight of 15-24 lb/1300 ft^2 (6.8 to 10.9 kg/121 m^2) is capable of running on a high speed laser printer or copier. When the paper is sized at an addition rate of at least 1.5 lb/ton (0.68 kg/0.9 metric ton, preferably at least 2.2 lb/ton (1 kg/0.9 metric ton), it is capable of running on the IBM model 3825 high-speed copier without causing misfeeds or jams at a rate of 5 or less in 10,000, preferably at a rate of 1 or less in 10,000. By comparison, paper sized with standard AKD has a much higher rate of double feeds on the IBM 3825 high speed copier (14 double feeds in 14,250 sheets). In conventional copy-machine operation, 10 double feeds in 10,000 is unacceptable. A machine manufacturer considers 1 double feed in 10,000 sheets to be unacceptable.

The paper of this invention in the form of a roll of continuous forms bond paper having a basis weight of 20-24 lb/3000 ft^2 (9.1 to 10.9 kg/279 m^2) can be converted to a standard perforated continuous form on a continuous forms press at a press speed of 1300 to 2000 feet (390 m to 600 m) per minute. The preferred paper according to the invention, in the form of a roll of continuous forms bond paper having a basis weight of 20-24 lb/3000 ft^2 (9.1 to 10.9 kg/279 m^2), and that is sized at an addition rate of at least 2.2 lb/ton (1 kg per 0.9 metric ton) can be converted to a standard perforated continuous form on the Hamilton-Stevens continuous forms press at a press speed of at least 1775 feet (541 m) per minute, preferably at least 1900 feet (579 m) per minute.

The paper of this invention can also be made into a roll of envelope paper having a basis weight of 20-24 lb/1300 ft^2 (9.1 to 10.9 kg/121 m^2) that is sized at an addition rate of at least 2.2 lb/ton (1 kg/0.9 metric ton). The paper can be converted into at least 900 envelopes per minute, preferably at least 1000 per minute on a Winkler & Dunnebier CH envelope folder.

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The paper of this invention can be run at a speed of at least 58 sheets per minute on a high speed IBM 3825 sheet-fed copier with less than 1 in 10,000 double feeds or jams.

The paper of this invention is capable of running on a high speed, continuous-forms laser printer with a rate of billowing at least 10% less, preferably 20% less, than that produced when running on the same printer, a roll of continuous forms bond paper having the same basis weight and sized at the same level with an AKD size made from a mixture of stearic and palmitic acids, after 10 minutes of running time.

The paper of this invention is capable of running on a high speed IBM 3825 sheet-fed copier at a speed of 58 sheets per minute with at least 50% fewer, preferably 70% fewer, double feeds or jams than the number of double feeds or jams caused when running on the same copier, sheets of paper having the same basis weight and sized at the same level with an AKD size made from a mixture of stearic and palmitic acids.

The paper of this invention is also capable of being converted to a standard perforated continuous form on a continuous forms press at a press speed at least 3% higher,

preferably at least 5% higher, than paper having the same basis weight and sized at the same level with an AKD size made from a mixture of stearic and palmitic acids.

Paper for evaluation on the IBM 3800 was prepared on a pilot paper machine. To make a typical forms bond paper-making stock, the pulp furnish (three parts Southern hardwood kraft pulp and one part Southern softwood kraft pulp) was refined to 425 ml Canadian Standard Freeness (C.S.F.) using a double disk refiner. Prior to the addition of the filler to the pulp furnish (10% medium particle-size precipitated calcium carbonate), the pH (7.8-8.0), alkalinity (150-200 p.p.m.), and hardness (100 p.p.m.) of the papermaking stock were adjusted using the appropriate amounts of $NaHCO_3$, NaOH, and $CaCl_2$.

The mixture of 2-oxetanone compounds was prepared by methods used conventionally to prepare commercial alkyl ketene dimers, i.e, acid chlorides from a mixture of a linear, saturated fatty acid and a linear, saturated alkyl dicarboxylic acid are formed, using a conventional chlorination agent, and the acid chlorides are dehydrochlorinated in the presence of a suitable base. Emulsions of the mixture of 2-oxetanone compounds were

prepared according to the disclosure of U.S. Patent 4,317,756, with particular reference to Example 5 of the patent. Wet-end additions of the 2-oxetanone multimer sizing agent, quaternary-amine-substituted cationic starch (0.75%), alum (0.2%), and retention aid (0.025%) were made. Stock temperature at the headbox and white water tray was controlled at 110°F (43.3°C).

The wet presses were set at 40 p.s.i. (2.8 kg/cm^2) gauge. A dryer profile that gave 1-2% moisture at the size press and 4-6% moisture at the reel was used (77 feet (23.4 m) per minute). Before the size press, the sizing level was measured on a sample of paper torn from the edge of the sheet, using the Hercules Size Test (HST). Approximately 35 lb/ton (15.9 kg/0.9 metric ton) of an oxidized corn starch and 1 lb/ton (0.45 kg/0.9 metric ton) of NaCl were added at the size press (130°F (54.4°C), pH 8). Calender pressure and reel moisture were adjusted to obtain a Sheffield smoothness of 150 flow units at the reel (Column #2, felt side up).

A 35 minute roll of paper was collected and converted on a commercial forms press to two boxes of standard 8 1/2" x 11" (21.6 x 28 cm) forms. Samples were also collected before and after each 35 minute roll for natural aged size testing

basis weight (46 lb/3000 ft^2, 20.8 kg/279 m^2), and smoothness testing.

The converted paper was allowed to equilibrate in the printer room for at least one day prior to evaluation. Each box of paper allowed a 10-14 minute (220 feet (66.7 m) per minute) evaluation on the IBM 3800. All samples were tested in duplicate. A standard acid fine paper was run for at least two minutes between each evaluation to reestablish initial machine conditions.

In order to establish whether a sizing agent contributes to difficulties in converting operations, paper was made on a pilot paper machine, converted into forms, and then printed on an IBM 3800 high speed printer. The runnability on the IBM 3800 was used as a measure of converting performance. Specifically, the height to which the paper billows between two defined rolls on the IBM 3800 was used to quantify converting performance. The faster and higher the sheet billows, the worse the converting performance.

The Hercules Size Test (HST) is a standard test in the industry for measuring the degree of sizing. This method employs an aqueous dye solution as the penetrant to permit optical detection of the liquid front as it moves through the

sheet. The apparatus determines the time required for the reflectance of the sheet surface not in contact with the penetrant to drop to a predetermined percentage of its original reflectance. All HST testing data reported measure the seconds to 80% reflection with 1% formic acid ink mixed with naphthol green B dye unless otherwise noted. The use of formic acid ink is a more severe test than neutral ink and tends to give faster test times. High HST values are better than low values. The amount of sizing desired depends upon the kind of paper being made and the system used to make it.

Example 1

This example describes the preparation of a mixture of 2-oxetanone compounds from a blend of stearic and sebacic acids at a molar ratio of 2.5.

A blend of stearic acid (227.2 g, 0.8 mole) and sebacic acid (64.64 g, 0.32 mole) with a molar ratio of 2.5 was heated to melt at a temperature in the range of 110° to 116°C under a nitrogen atmosphere. The molten acid blend was then poured into a pyrex jacketed vessel equipped with a mechanical stirrer, condenser, nitrogen purge, and thermocouple and preheated to 95°C. When the molten acids in

the vessel cooled down to a temperature of 75° to 85°C, PCl_3 (130.5 g) was dropped into the reactor all at one time. After 3 hours of chlorination and settling, phosphorous acid was separated from the acid chloride blend. The excess PCl_3 was stripped off under a vacuum to recover the acid chloride blend (307.5 g, 1.12 moles) in an almost theoretical yield. The blend of stearic and sebacic chlorides was then added by drops to a reactor containing propylene dichloride as the reaction solvent (650 ml) and triethylamine (160 g, 1.584 moles,10% molar excess) as the base catalyst at 40° to 45°C. After 2 hours, the triethylamine hydrochloride salt was separated by suction filtration and a stearic/sebacic 2-oxetanone multimer mixture was recovered as a pale-yellow solid (232.78 g). Solvent and excess triethylamine were evaporated to give an overall yield of 90% multimer. The mixture contained 54.7% multimers having 2 or more oxetanone rings.

Example 2

This example describes the preparation of a mixture of 2-oxetanone compounds from a blend of palmitic/azelaic acids at a molar ratio of 2.5.

A blend of palmitic acid (204.8 g, 0.8 mole) and azelaic acid (60.16 g, 0.32 mole) with a molar ratio of 2.5 was heated to melt at a temperature of 80° to 85°C under a nitrogen atmosphere. The molten acid blend was then poured into a pyrex glass jacketed vessel equipped with a mechanical stirrer, condenser, nitrogen purge and thermocouple, and preheated to the chlorination temperature (70±2°C). The chlorinating agent, PCl_3 (97.9 g) was added to the vessel all at one time. The vessel temperature dropped 2° to 5°C initially, but eventually stabilized at 70±2°C. After 3 hours of chlorination and settling, phosphorous acids were separated from the acid chloride blend. The excess PCl_3 was stripped off under a vacuum to recover 290.0 g of the acid chloride blend in an almost theoretical yield. The blend of palmitic and azelaic chlorides was then added by drops to a reactor containing propylene dichloride (600 ml) as the reaction solvent and triethylamine (160 g, 10% excess) as the base catalyst at 40° to 45°C. After two hours, the triethylamine hydrochloride salts were separated by suction filtration and the 2-oxetanone multimer mixture was recovered as a pale-yellow solid by evaporation of solvent and excess triethylamine. The an overall yield was 96% (240.53 g). The

mixture contained 58.2% multimers having 2 or more oxetanone rings.

Example 3

In this example a number of sizing agents were tested for their effects on the IBM 3800 runnability of a difficult-to-convert grade of alkaline fine paper. The control was a standard AKD sizing agent made from a mixture of stearic and palmitic acids using the method described in Examples 1 and 2. The mixture of 2-oxetanone compounds used in samples 2-6 was prepared using the method described in Examples 1 and 2, using the following mixtures of mono- and dicarboxylic acids at the mole ratio indicated in the table: Sample 2 - palmitic acid/dodecanedioic acid; Sample 3 - palmitic acid/sebacic acid; Sample 4 - palmitic acid/azelaic acid; Sample 5 - stearic acid/sebacic acid; Sample 6 - stearic acid/EMPOL 1008 C_{36} dimer acids, available from Henkel-Emery, Cincinnati, Ohio, U.S.A. The percentage of 2-oxetanone compounds containing two or more rings is: Sample 2, 58.2%; Sample 3, 64.7%; Sample 4, 58.2%; Sample 5, 54.7%, and Sample 6, 63.1%.

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The results of the sizing and converting tests are given in Table 1.

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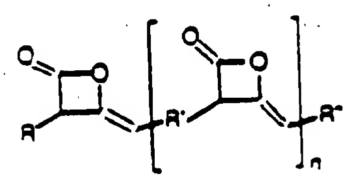
TABLE 1

Sample	Description	Size Addition Rate kg/0.9 metric ton	On Machine HST (sec)	7 Day HST (sec)	Time to 3" (7.6 cm) Billowing (sec)	Maximum Billow (cm)	Time to 4" (10.2 cm) Billowing (sec)
1 (Control)	Stearic/palmitic acid ketene dimer	1	231	295	180	8.6	>765
2	2.5:1 molar ratio of C_{16}/C_{12di} 2-oxetanone multimer	1	186	248	>705	6.4	never
3	2.0:1 molar ratio of C_{16}/C_{10di} 2-oxetanone multimer	1	183	243	>850	5.4	never
4	1:1 molar ratio of C_{16}/C_{9di} 2-oxetanone multimer	1	115	218	>760	5.4	never
5	2.0:1 molar ratio of C_{18}/C_{10di} 2-oxetanone multimer	1	150	220	>720	7.6	>760
6	1.75:1 molar ratio of C_{18}/C_{36di} 2-oxetanone multimer	1	20	98	>700	5.1	never

The data show that the 2-oxetanone sizing agents of this invention gave a better balance of sizing and converting performance (less billowing at the same level of sizing) than the commercial alkene dimer size used as the control. The best balance of sizing and handling performance was obtained with Sample 3. This sizing agent gave a level of sizing comparable to that obtained with the AKD control and gave paper with better runnability on the IBM 3800 than the paper sized with the AKD control.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:-

1. Paper sized with a sizing agent comprising a mixture of 2-oxetanone compounds, characterized in that the paper is made under alkaline conditions and the 2-oxetanone compounds have the formula



in which n is 0 or an integer; R and R" can be the same or different and are saturated, linear alkyl groups having 8-24 carbon atoms; R' is a saturated, linear alkyl group having 4-40 carbon atoms, and n = at least one in at least 50% by weight of the compounds in the mixture, and the paper does not encounter significant machine-feed problems on high speed converting machines or in reprographic operations.

2. The paper of claim 1, further characterized in that R and R" are saturated, straight chain alkyl groups having 14 or 16 carbon atoms.

3. The paper of claims 1 or 2, further characterized in that R′ is a saturated, straight chain alkyl group having 5-6 carbon atoms.

4. The paper of any of the preceding claims, further characterized in that the percentage of 2-oxetanone compounds in the mixture having n = at least one is at least 60% by weight.

5. The paper of any of the preceding claims, further characterized in that the size additionally comprises a water soluble inorganic salt of an alkali metal.

6. The paper of claim 5, further characterized in that the alkali metal salt is sodium chloride.

7. The paper of any of the preceding claims, further characterized in that the paper is externally sized.

8. The paper of claims 1-6, further characterized in that the paper is internally sized.

9. The paper of any of the preceding claims, further characterized in that the sizing agent is made from palmitic or stearic acid, and azelaic acid.

10. The paper of claims 1-9, further characterized in that it is in the form of continuous forms bond paper.

11. The paper of claims 1-9, further characterized in that it is in the form of adding machine paper.

12. The paper of claims 1-9, further characterized in that it is in the form of envelope-making paper.

13. The paper of claims 1-9, further characterized in that it is in the form of an envelope.

14. The process of passing the paper of claims 1-9 in the form of a roll of continuous forms bond paper having a basis weight of 30-60 lb/3000 ft^2 (13.6 to 27.2 kg/279 m^2) through a high speed, continuous-forms laser printer.

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15. The process of claim 14, further characterized in that the paper had been sized at a size addition rate of at least 2.2 lb/ton (1 kg/0.9 metric ton) and the process is carried out without causing a rate of billowing in inches of increase per second x 10,000 greater than 5 after 10 minutes of passing the paper through an IBM Model 3800 high speed, continuous-forms laser printer.

16. The process of passing the paper of claims 1-9 in the form of 8½ x 11 inch (21.6 x 28 cm) reprographic paper having a basis weight of 15-24 lb/1300 ft^2 (6.8 to 10.9 kg/121 m^2) through a high speed laser printer or copier.

17. The process of claim 16, further characterized in that the paper had been sized at an addition rate of at least 2.2 lb/ton (1 kg/0.9 metric ton) and the paper is passed through on an IBM 3825 high speed copier without causing misfeeds or jams at a rate of 5 or less per 10,000 sheets.

18. A process for converting the paper of claims 1-9 in the form of a roll of continuous forms bond paper having a basis weight of 20-24 lb/3000 ft^2 (9.1 to 10.9 kg/279 m^2) to a

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standard perforated continuous form which comprises passing the paper through a continuous forms press at a press speed of 1300-2000 ft (390 m to 600 m) per minute.

19. A process for converting the paper of claims 1-9 in the form of roll of continuous forms bond paper having a basis weight of 20-24 lb/3000 ft^2 (9.1 to 10.9 kg/279 m^2) and having been sized at an addition rate of at least 2.2 lb/ton (1 kg/0.9 metric ton) to a standard perforated continuous form which comprises passing it through a Hamilton-Stevens continuous forms press at a press speed of at least 1775 feet (532 m) per minute.

20. A process for converting the paper of claims 1-9 in the form of a roll of envelope paper having a basis weight of 20-24 lb/1300 ft^2 (9.1 to 10.9 kg/121 m^2) having been sized at an addition rate of at least 2.2 lb/ton (1 kg/0.9 metric ton) into envelopes which comprises passing the paper through a Winkler & Dunnebier CH envelope folder at a rate sufficient to produce 900 envelopes per minute.

21. The paper of claims 1-9, further characterized in that it is capable of running on a high speed, continuous-forms laser printer with a rate of billowing at least 10%

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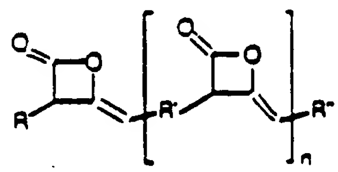
less than that produced when running on the same printer, a roll of continuous forms bond paper having the same basis weight and sized at the same level with an AKD size made from a mixture of stearic and palmitic acids, after 10 minutes of running time.

22. The paper of claims 1-9, further characterized in that it is capable of running on a high speed IBM 3825 sheet-fed copier at a speed of 58 sheets per minute with at least 50% fewer double-feeds or jams than the number of double-feeds or jams caused when running on the same copier, sheets of paper having the same basis weight and sized at the same level with an AKD size made from a mixture of stearic and palmitic acids.

23. The paper of claims 1-9, further characterized in that it is capable of being converted to a standard perforated continuous form on a continuous forms press at a press speed at least 3% higher than paper having the same basis weight and sized at the same level with an AKD size made from a mixture of stearic and palmitic acids.

24. A mixture of 2-oxetanone compounds characterized in that the 2-oxetanone compounds have the formula



in which n is 0 or an integer; R and R" can be the same or different and are saturated, linear alkyl groups having 8-24 carbon atoms, and R′ is a saturated, linear alkyl group having 4-40 carbon atoms, said mixture being the reaction product of a reaction mixture comprising at least one 10-26 carbon linear, saturated alkyl monocarboxylic acid and at least one 8-44 carbon linear, saturated alkyl dicarboxylic acid, wherein the ratio of mono- to dicarboxylic acid is 1.0 to 3.5, and at least 50% by weight of the compounds in the mixture have at least two 2-oxetanone rings.

25. The mixture of claim 24, further characterized in that the mole ratio is 2.5.

26. The mixture of claim 25, further characterized in that the mole ratio is 2.0.

27. The mixture of claims 24-26, further characterized in that the dicarboxylic acid is selected from the group consisting of a 9-10 carbon, 22 carbon, and 36 carbon dicarboxylic acid.

28. The mixture of claim 27, further characterized in that the dicarboxylic acid has 9-10 carbon atoms.

29. The mixture of claims 24-28, further characterized in that the monocarboxylic acid has 14-22 carbon atoms.

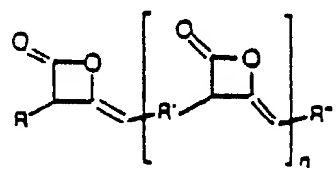
30. The mixture of claim 29, further characterized in that the monocarboxylic acid has 16 - 18 carbon atoms.

31. The mixture of claim 29, further characterized in that the monocarboxylic acid is a mixture of 16 carbon and 18 carbon linear, saturated alkyl monocarboxylic acids.

32. The mixture of claims 24-31, further characterized in that at least 60% by weight of the compounds have at least two 2-oxetanone rings.

33. The mixture of claims 24-30, further characterized in that the monocarboxylic acid is palmitic or stearic acid, and the dicarboxylic acid is azelaic acid.

34. A process for making paper for performance in precision converting or reprographic machinery, characterized by the step of internally sizing the paper under alkaline conditions with a 2-oxetanone sizing agent comprising a mixture of 2-oxetanone compounds having the formula:



in which n is 0 or an integer, R and R" can be the same or different and are saturated, linear alkyl groups having 8 - 24 carbon atoms; R' is a saturated, linear alkyl group having 4 - 40 carbon atoms, and n = at least one in at least 50% by weight of the compounds in the mixture.

35. Use of paper sized with a sizing agent comprising a mixture of 2-oxetanone compounds, characterized in that the

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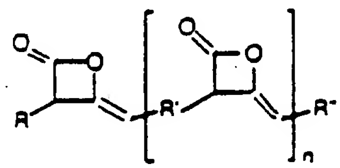
paper is sized under alkaline conditions; the 2-oxetanone compounds having the formula



in which n is 0 or an integer, R and R" can be the same or different and are saturated, linear alkyl groups having 8 - 24 carbon atoms; R' is a saturated, linear alkyl group having 4 - 40 carbon atoms, and n = at least one in at least 50% by weight of the compounds in the mixture, and the paper is used in high speed converting or reprographic operations.

36. The process of claim 34, characterized in that R and R" have 14 or 16 carbon atoms.

37. The process of claim 34 or claim 36 characterized in that R' has 5 - 6 carbon atoms.

38. The use of claim 35, characterized in that R and R" have 14 or 16 carbon atoms.

39. The use of claim 36 or claim 38 characterized in that R' has 5 - 6 carbon atoms.